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Improved RRR measurement procedure at the FNAL TD SSTF and observations on the sample holder material

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This note describes a better procedure for measuring the residual resistivity ratio (RRR) of the copper stabilizer of superconducting strands at the Short Sample Test Facility (SSTF), and shows how much the conductivity of the Ti-6Al-4V sample holder affects these measurements. The new procedure allows reducing sensibly the temperature gradient on the strand during the measurement in order to achieve higher precision and allows estimating the critical temperature (T_C) of the superconductor. The analysis on the Ti-6Al-4V barrel shows that its conductivity introduces a large error in RRR measurement.

I. Introduction

RRR of copper is the ratio between the electrical resistivity at 300K temperature and that at the temperature of the boiling helium 4.2 K. At the SSTF, in order to evaluate the RRR of the copper in SC strands, the resistivity at 4.2K is estimated by measuring the resistance of the strand at a temperature just above the superconductor T_C. During this measurement a known current is supplied and the barrel (fig. 1), on which the strand is wound, is assumed to have an infinite resistance. Voltage signals are taken through two pairs of taps on the strand respectively 50 and 75 cm apart from each other. The conductivity of the non-copper area is assumed negligible while the copper resistivity at room temperature is assumed equal to $1.67 \cdot 10^{-8}\ \Omega\ m$.

Using this technique, relevant causes of errors in the evaluation of RRR, are:

1) non-uniform temperature of the sample;
2) current sharing between the SC wire and the barrel (the barrel is in direct contact with the copper leads and the strand).

These two problems have been analyzed in the following two paragraphs.

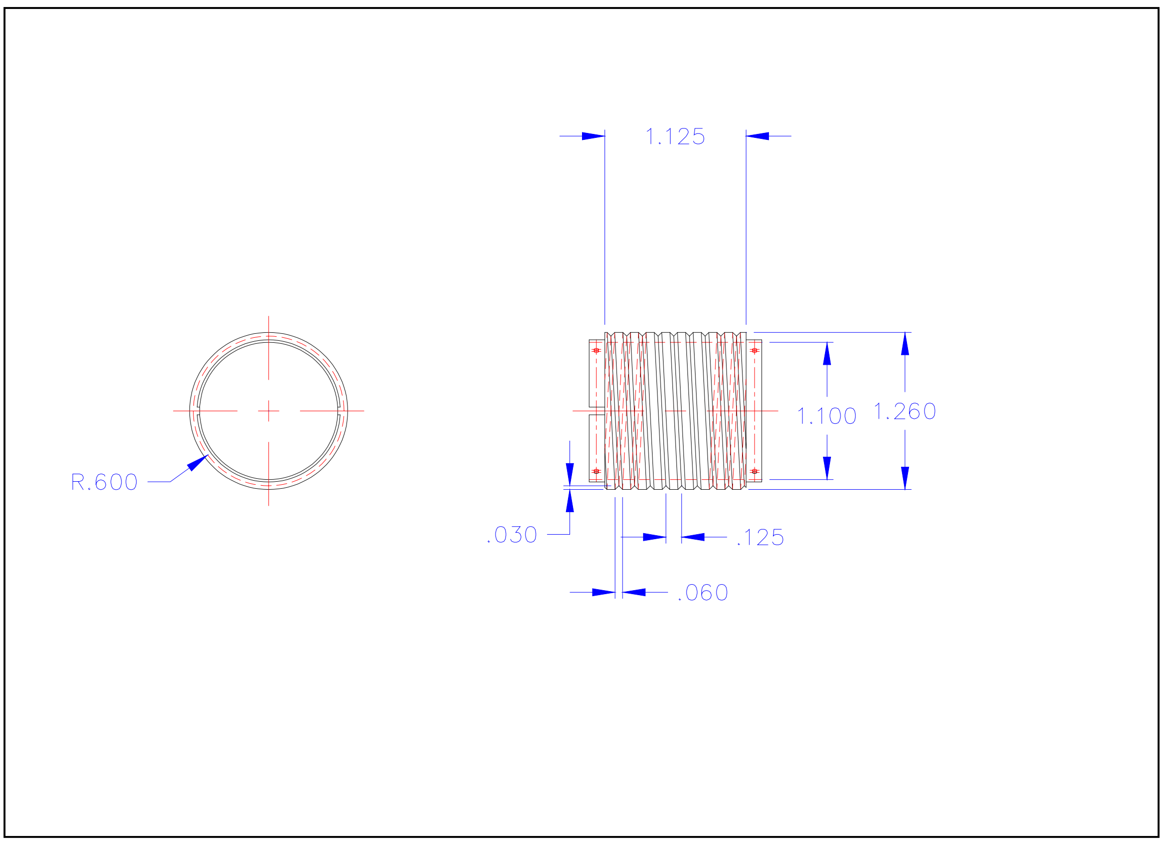

Fig. 1. Barrel geometry (dimension in inch).

II. Thermal observations and upgraded measurement procedure

The resistance at low temperature is measured by warming up the whole sample to a temperature just above the superconductor T_c. The increase of temperature must be controlled not to overcome the copper plateau. For example, when the copper RRR is 20 the resistivity starts increasing sensibly with temperature at 20 *K* (fig. 2). According to the previous procedure this condition is achieved by closing the needle valve to stop the liquid helium flow in the VTI and then by turning on the heater to evaporate the helium and to warm up the sample. The strand resistance is measured during the warm up. In order to confirm the results, the measurement is repeated during the cool down of the sample by opening the needle valve. Fig. 3 shows a typical result of this procedure. The main point to observe is that, during the warm up, the transition temperature recorded by the cernox is several degrees lower than the one obtained during the cool down. This cernox records the temperature of the helium gas at the height of the sample, that means the gas temperature is lower than the sample one when warming up and higher when cooling down. As confirmed by the readings of the cernox on the bottom of the VTI fig.3, the vessel that contains the probe, this behavior is due to the fact that the gas temperature is stratified. This axial thermal non-uniformity depends on the fact that the sample is warmed and cooled from the bottom where there are the heaters and the entrance of liquid helium.

Thermal non uniformity doesn't allow correlating the resistance measurement of the strand with temperature: the sample temperature can be different point to point and the cernox reading can be different from the average temperature of the sample as fig. 3 shows.

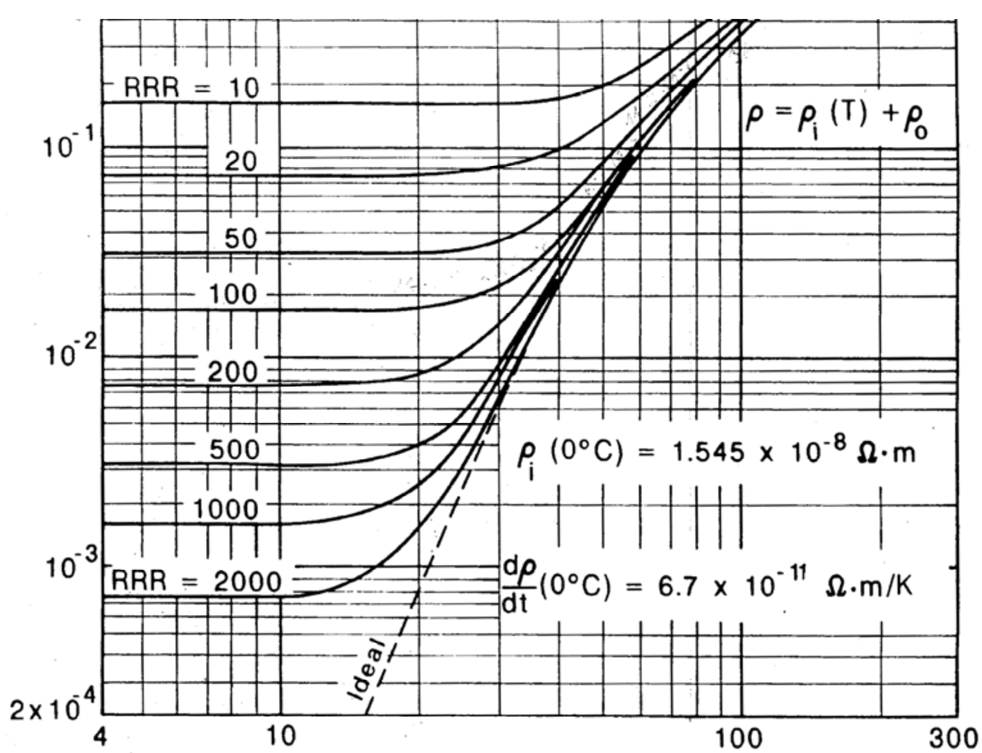

Fig. 2. Copper resistance.

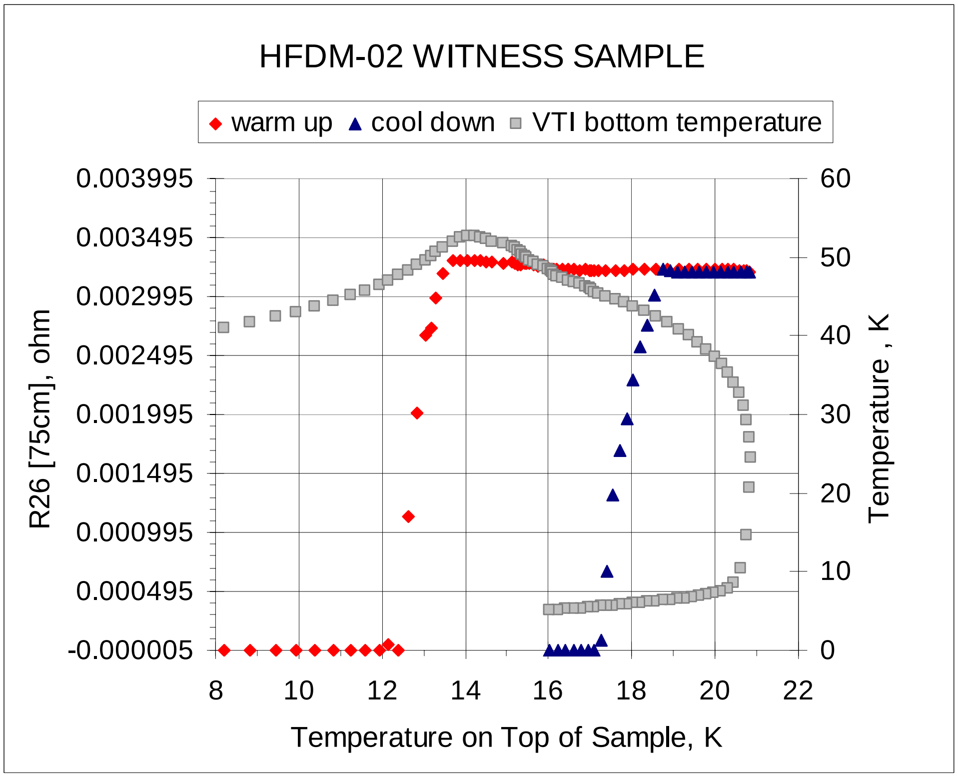

Fig. 3. RRR measurement with the old procedure.

In RRR measurement this is a problem when the copper plateau is at a lower temperature than Tc. This can be the case of Nb_3Sn strands where the critical temperature of the superconductor is about 18 *K*.

As example of errors that thermal non uniformity can introduce fig. 4 and 5 show the resistance measurements of the same strand respectively with a uniform (low difference between the temperatures recorded by the two cernox during the whole measure) and non-uniform temperature atmosphere around the sample. This strand has a RRR of about 10 and was wounded on a G-10 barrel. While with uniform conditions, fig 4, the resistance between the transition temperature and 20*K* increase from 6.5 to 6.6 *m Ω*, with non uniform conditions, fig.5, it goes from 5.9 to 6.2 *mΩ*. In this case, using non-uniform conditions leads to an overestimate of the RRR of about 10%. The higher the RRR, the more relevant this kind of problem can be, since the copper plateau is at lower temperature.

The upgraded procedure is based on the idea that a strong helium gas flow around the sample and the leads allows stabilizing and uniforming the temperature. This condition is obtained by vaporizing the whole helium in the VTI and then opening the needle valve and turning on the heater. While the needle valve has to be opened as much as possible, to maintain the maximum gas flow, the heater power has to be regulated to reach the thermal equilibrium with the whole probe surrounded by vaporized helium. This condition is achieved when the readings of the temperature on the strand and on the bottom of the probe are constant and higher than 4.2*K*. Once the thermal equilibrium is reached the power of the heaters must be adjusted to modify the sample temperature.

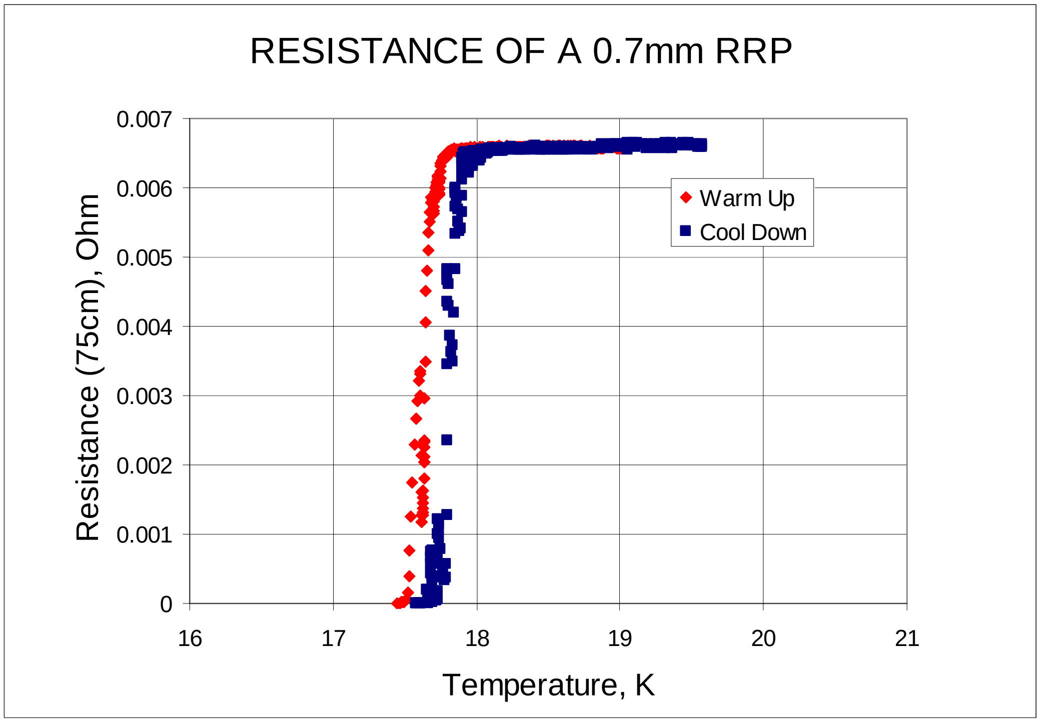

Fig. 4. RRR measurement with a good thermal uniformity in the sample.

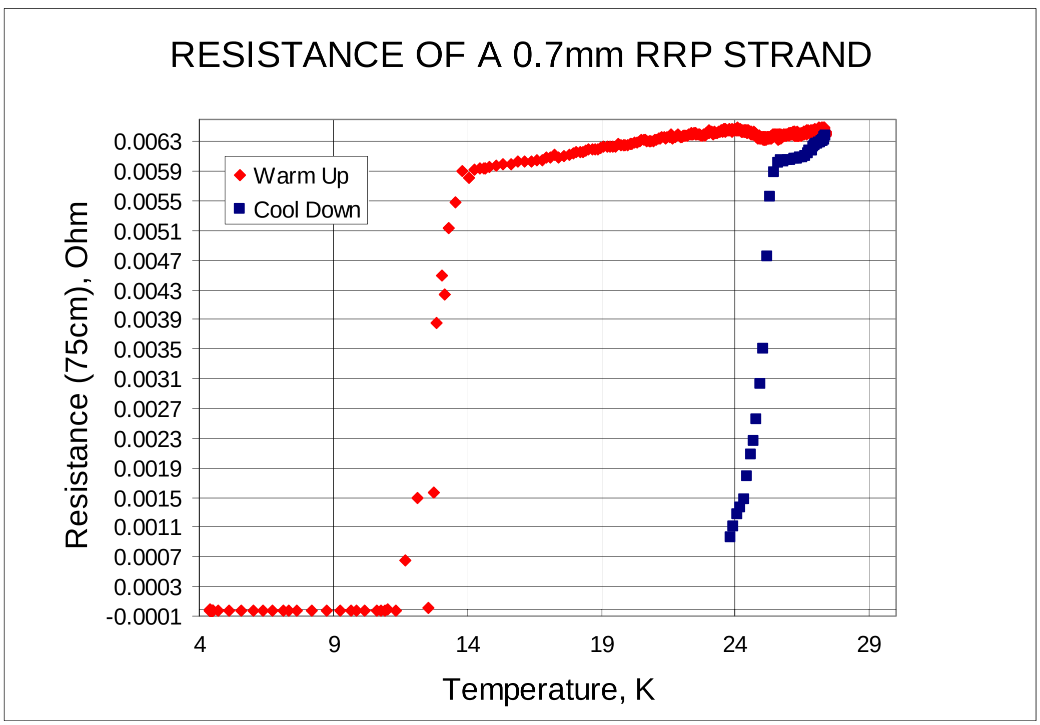

Fig. 5. RRR measurement with thermal non-uniformity in the sample.

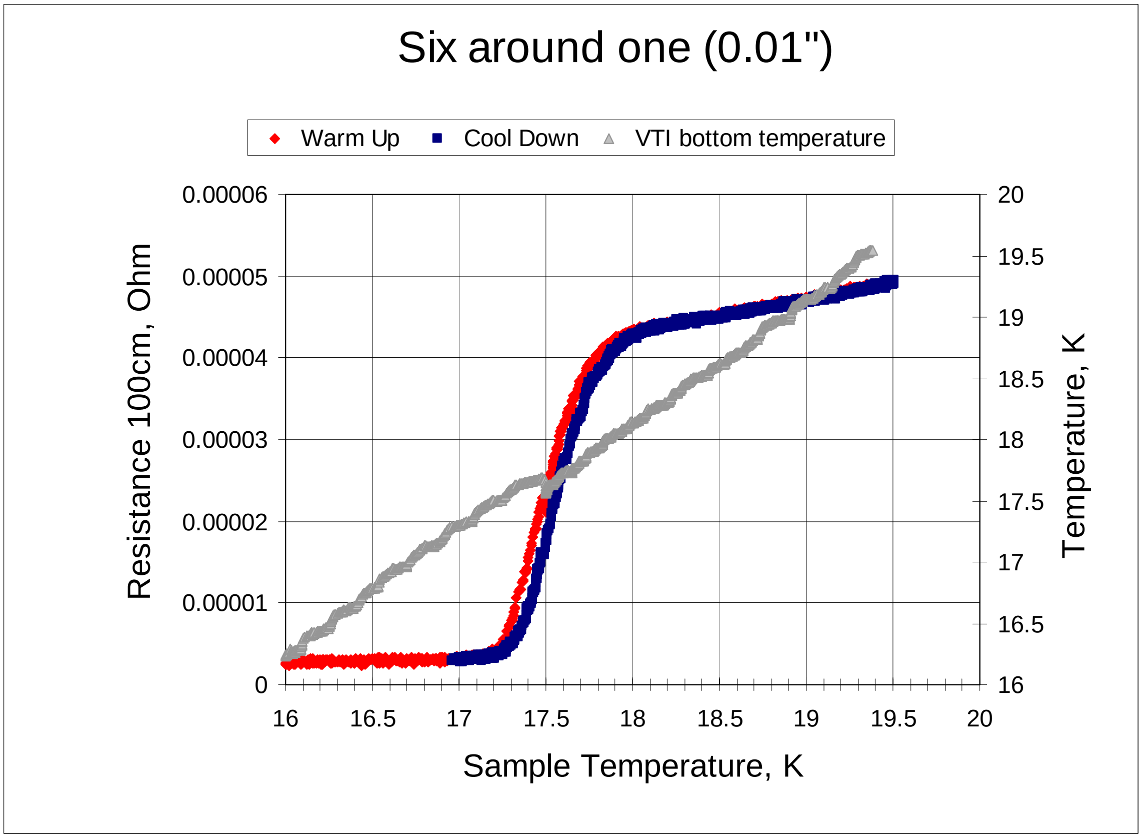

Fig. 6. RRR measurement with the new procedure.

An example of using this new procedure is shown in fig. 6 for a Nb_3Sn six-around one strand. The measured strand resistance is almost the same during the warm up and cool down of the sample: at the same value of resistance the difference in temperature is lower than 0.06*K*. With this upgraded procedure the temperature distribution in the sample is uniform, thus avoiding errors related to thermal non uniformity in RRR measurement. In addition, the critical temperature of the superconductor can be estimated within an error of ±0.05K

III. Estimate of the error on RRR measurements due to barrel material.

The purpose of the following analysis is to show why the alloy used to build the barrel (Ti-6Al-4V) for critical current measurement is not a suitable material for measuring the RRR and can be a reason for the large error obtained in such measurement. While measuring the strand resistance, it is assumed that all the current flows only through the strand, but due to the low barrel resistance, a consistent amount passes through the sample holder as well. In order to correctly evaluate the amount of current that flows through the barrel, it is necessary to define: the contact resistance between the strand, the current leads and the barrel; and the resistance of these two components.

Since an accurate analysis of the current sharing between the strand and the holder involves variables that at present are not measured (contact resistance) during RRR measurements, only two representative and significant cases will be discussed here as a demonstration of the effect on the holder material on the measurement. In the first configuration it is assumed that the contact resistance between the current leads and the barrel is negligible: this case is representative of the maximum error that can be committed in the evaluation of RRR. The second configuration is based on data measured during two tests of a NbTi strand with a high resistive matrix (Cu30Ni). These two experiments were performed one with a Ti-6Al-4V barrel and the other with a G-10 barrel and allow us to estimate the overall (contact + material) resistance of the Ti-6Al-4V barrel. Using this resistance it is possible to estimate the average error committed using the Ti-alloy barrel.

<u>Case 1: Negligible contact resistance</u>

In this configuration the barrel and the strand act like two parallel circuits where the resistance of each circuit is only due to the respective geometry and resistivity of the two components. Thus, to evaluate the current sharing, it is necessary to define the resistance of the barrel and of the strand at about 20 *K* as shown in Eq. 1.

$$R_S I_S = R_B I_B \longrightarrow \boxed{\frac{I_B}{I_S + I_B} = 1 - \frac{R_B}{R_B + R_S}} \qquad \textit{eq. 1}$$

S ≡ Strand

B ≡ Barrel

Without taking into account the Ti-6Al-4V SC behavior, the electrical resistivity of this alloy is 1.47 $\mu\Omega \cdot m$ @ 4*K* and 1.78 $\mu\Omega\cdot$m @ 300*K*. Using these values and the geometry shown in fig. 1 one can estimate that the resistance for a current flowing in the axial direction of the barrel, is about 250 $\mu\Omega$. This value is consistent with data (220 $\mu\Omega$) published in literature for similar barrels[1] and with a 4 wire measurement performed at room temperature (200-300 $\mu\Omega$).

On the barrel ~ 1 m of SC strand is wound. Its resistance, at temperature just above T_c, assuming a diameter of 1*mm* with 50% of copper is: $R_S = 4.25\cdot10^{-2}/\mathrm{RRR}\ \Omega$. So according to eq. 1:

$$\frac{I_B}{I_S + I_B} = 1 - \frac{250}{250 + \frac{4.25\cdot10^4}{RRR}}$$

For a strand with RRR=100 the fraction of the current that flows in the barrel is about 63% and the RRR estimated through our procedure (RRR_e) is:

RRR_e= 100/(1-0.63)=270

The case of negligible contact resistance between the copper leads and the barrel is an extreme condition that, even without considering the SC behavior of the Ti-alloy, leads to an error of 270% in the RRR measurement when RRR is 100. As shown in eq. 1 the lower the RRR the higher is the error.

As it will be shown in case two, the contact resistance plays an important role that reduces the error of the measurements. However this case is still indicative of the uncertainty introduced in RRR measurement using a conductive barrel.

Case 2: Overall barrel resistance evaluated through experimental data

Measurement of a NbTi strand with a high resistivity matrix (Cu30Ni) performed with a Ti-6Al-4V barrel (fig. 7) and with a G-10 barrel (fig. 8) allowed evaluating the overall (contact + material) resistance of the Ti-alloy barrel.

At a temperature higher than Tc, the resistance of the strand is measured to be 50 times higher than the resistance the Ti-alloy barrel + strand (fig. 7 and 8), confirming strand specifications.

Looking at fig. 7 one can then conclude that the overall resistance of the Ti-6Al-4V barrel for this experiment is about 7.3 *mΩ* and, since the resistance of the Ti-alloy barrel alone is lower than 0.3 *mΩ*, this value is mainly due to the contact resistance.

In this case the current ratio for the Ti-alloy barrel wound with the same strand used in case 1 is:

$$\frac{I_M}{I_S + I_M} = 1 - \frac{7300}{7300 + \dfrac{4.25 \cdot 10^4}{RRR}} \qquad \textit{eq. 2}$$

Using the above relation, one obtains Table I that shows the ratio of current flowing in the Ti-alloy barrel, the RRR estimated through our procedure and the error committed as a function of the strand RRR. For RRR lower than 10, typical of HF magnets produced at FNAL, the error predicted by Eq. 2 is very high. The order of magnitude of this prediction is consistent with the comparison between RRR data measured at VMTF, and RRR data measured at SSTF.

For example the RRR of the HFDM 02 measured at VMTF is 6.3 [2] while at the SSTF the sample showed in fig. 3 was measured to be 10.6. This means that the error committed is 68% and for strand with RRR between 5 and 10 tab. 1 foresees an error between 116.4 and 58.2%.

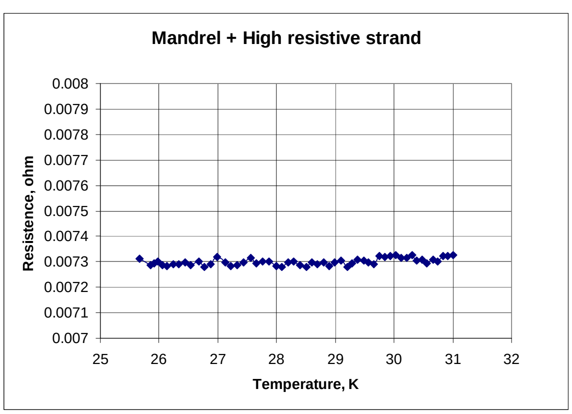

Fig. 7 Ti-6Al-4V barrel coiled with a high resistive matrix NbTi strand

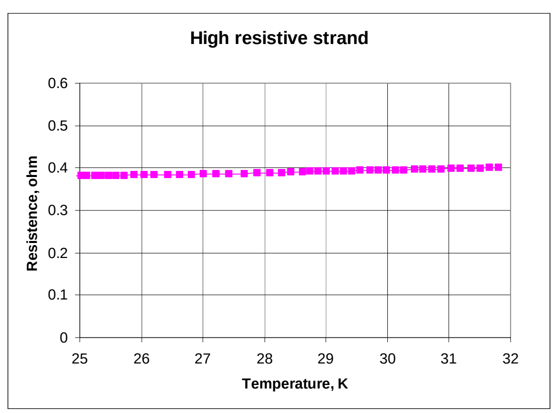

Fig. 8 G-10 barrel coiled with a high resistive matrix NbTi strand

An additional RRR test was performed on two RRP strands extracted from the same cable, coming from the same billet and reacted at the same time. One strand was measured on the Titanium barrel and the other on the G-10 barrel. The results for the Titanium is 16.5 while for the G-10 is 11.7. These results are in agreement with Table I.

This case 2, summarized by tab.1, shows that the error in the estimate of the copper RRR in a SC strand at SSTF while using Ti barrel, can be very high.

The error prediction presented in the table can not be used for an accurate correction of the measurement performed at SSTF because the overall resistance of the Ti-alloy barrel may vary sensibly test by test due to different contact resistance with the strand and the copper leads.

This difference in contact resistance can be the reason of the spread of RRR data at SSTF.

RRR	**barrel current ratio**	**RRRe**	**error %**
5	0.538	10.8	116.4
10	0.368	15.8	58.2
20	0.225	25.8	29.1
30	0.163	35.8	19.4
40	0.127	45.8	14.6
50	0.104	55.8	11.6
60	0.088	65.8	9.7
70	0.077	75.8	8.3
80	0.068	85.8	7.3
90	0.061	95.8	6.5
100	0.055	105.8	5.8
120	0.046	125.8	4.9
140	0.040	145.8	4.2
160	0.035	165.8	3.6
180	0.031	185.8	3.2
200	0.028	205.8	2.9

Table I. RRR estimated (RRR_e) at SSTF with a barrel overall resistance of 7.3 $m\Omega$

IV. Conclusions

Analyzing the RRR measurement procedure and results at SSTF one can deduce that the temperature of the gas atmosphere around the sample is not uniform. Thermal non uniformity doesn't allow correlating the resistance measurement of the strand with temperature. In RRR measurement of Nb_3Sn strands this can lead to errors since the copper plateau can be at a lower temperature than Tc. In the test showed above, for a strand with a RRR of about 10, non-uniformity leads to overestimate the RRR of about 10%.

In order to solve this problem a new effective measurement procedure has been created. Results show that during the sample warm up and cool down the measured resistance are practically the same with this new procedure. Beyond improving RRR measurement, the major result of this procedure is that it allows estimating the critical temperature of the superconductor within an error of ±0.05K.

As far as barrel material it is found that the Ti-6Al-4V conductivity lets a non-negligible amount of current to flow through it. This leads to errors in RRR measurement up to a factor of two. Moreover this error can not be predicted with accuracy due to the non repeatability of contact resistance between the barrel and the current leads (SC strand and copper leads). The non repeatability of contact resistance is a possible substantial contribution of the significant spread of RRR data when measured on Ti-alloy barrels. An additional likely contribution to the spread in the data is the non homogeneity of the material.

In order to improve the accuracy of RRR measurement without changing barrel material it would be useful to: 1) increase the contact resistance between the barrel and the current leads; 2) monitoring the voltage in the barrel to estimate the current that flows through it.

Samples used as magnet witnesses used to be all measured on Ti-alloy barrels as opposed to G-10 barrels in order to provide an acceptable statistics on Ic measurements. These are important to predict magnet short sample limits, and measurements on G-10 barrels are not representative of the actual critical current. However, given the recent understanding of the relevance of the RRR, when a magnet is reacted it is worth to include a couple of witness samples meant to be transferred after reaction to G-10 barrels for more precise RRR measurements.

References

1 Vol 5,N 3,September 1995 IEEE TRANSACTION ON APPLIED SUPERCODUCTIVITY

2 S. Feher et al. 28, August 2003,"HFDM 02 test summary", FNAL TD-03-041